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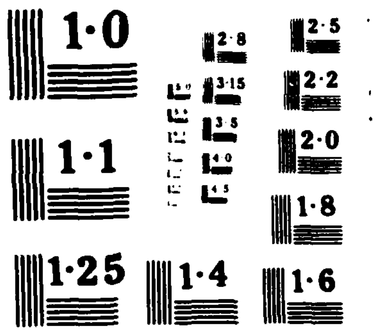
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EFFECT OF POST-CURE HEAT-TREATMENT ON STATIC AND FATIGUE BEHAVIOUR OF XAS-914C CARBON FIBRE COMPOSITE (U)

by

T.J. van Blaricum and G. Clark

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XAS 914C carbon fibre composite requires a post cure heat treatment cycle to complete polymer cross linking in the epoxy matrix material. The effect of omitting the post cure cycle on the fatigue performance of 56 ply laminate containing impact damage and on the static compressive strength of the same laminate was investigated.

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by

T.J. van Blaricum and G. Clark

SUMMARY

XAS-914C carbon fibre composite requires a post-cure heat-treatment cycle to complete polymer crosslinking in the epoxy matrix material. The effect of omitting the post-cure cycle on the fatigue performance of 56-ply laminate containing impact damage and on the static compressive strength of the same undamaged laminate was investigated.

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1. INTRODUCTION

In the production of aircraft structural components from carbon fibre composite (CFC) materials, the finished laminates, consisting of a number of plies, are cured in an autoclave using a temperature/pressure cycle prescribed by the manufacturer of the preimpregnated (prepreg) unidirectional ply sheet. In the case of XAS-914C (a CFC material currently being used by ARL for investigation of structural fatigue), as with most CFC materials, a post-cure cycle is also required. The function of this post-cure cycle (Appendix 1) is to complete the polymer crosslinking process in the epoxy matrix material, thereby optimizing the mechanical properties of the cured laminate. In a laboratory environment it should be possible with the correct equipment to follow precisely the manufacturer's recommended cure cycle. In a production environment, however, where there is a requirement to maximize efficiency, it may be necessary to use a compromise cure cycle, so that components laid up using various prepreg systems or incorporating adhesive bonds which are to be co-cured can be cured in the same autoclave. In a production environment, therefore, there exists the possibility that some layups may not be fully cured if the compromise cure cycle is not designed correctly. This technical memorandum summarises observations made on the influence of post-cure heat treatment on the compression-dominated fatigue behaviour of impact-damaged 56-ply XAS-914C coupons. These coupons were used primarily to investigate the feasibility of accelerating the fatigue testing of composite material structures (Ref 1) by the deletion of low load excursions from a flight-by-flight sequence. Also described are the results of static compression strength tests on unimpacted (undamaged) specimens cut from the ends of the fatigue test coupons.

2. FATIGUE TESTS

A brief summary of the fatigue test procedure and specimens is presented below; details are given in Ref. 1.

2.1. Fatigue Test Coupons

The 300 mm x 100 mm fatigue test coupons (Fig. 1a) with the 0^{o} fibre direction along the length of the coupon, were cut from three 56-ply XAS-914C panels (designated FG, FH and FI) with layup $(\pm 45/0_2)_{7S}$. These panels were manufactured at Aeronautical Research Laboratories (ARL), according to the procedures (Appendix

1) recommended by Ciba Geigy. The thickness of the cured coupons was approximately 7 mm.

2.2. Fatigue Test Procedure

All coupons were impacted in the centre of the gauge area. A rigid clamp with a circular window was used to restrict impact damage to a maximum diameter of a 30 mm. The coupons were tested under the influence of either a standard or a truncated FALSTAFF loading sequence with the zero degree fibre direction parallel to the loading axis of a 500 kN Instron fatigue testing machine; details are given in Ref. 1. The maximum gross area strain for all fatigue tests was 3750 microstrain.

2.3. Fatigue Test Results

Fatigue test coupon lives are shown in Table 1, together with the results of a statistical analysis which revealed that, while the load spectrum modification used had no significant effect on the specimen fatigue life, panel-to-panel variations did have a significant effect. Examination of the coupon material by Thermo Mechanical Analysis revealed that panel FG displayed a state of cure considerably less advanced than was the case for the other two panels; it was concluded that the post-cure heat treatment of panel FG had probably been omitted. Coupons from this panel, as can be seen from Table 1, gave significantly lower coupon fatigue lives.

3. STATIC COMPRESSION TESTS

3.1. Static Compression Test Coupons

To investigate whether the static compression strength of unimpacted CFC material from panel FG was also degraded due to the lack of post curing, a total of twenty-eight test coupons of the dimensions shown in Fig. 1b, were cut from the available undamaged ends of some of the original 300 mm x 100 mm fatigue test coupons. This provided 10 coupons from panel FG and nine coupons from each of panels FH and FI. Fully random selection of coupons from the three original uncut panel areas was not possible, as some fatigue test coupon ends had been used for impacting trials. In some cases, up to four static test coupons from one pair of ends had to be utilized to obtain a reasonable sample size. Where this was necessary the coupons were given the suffix A, B, C or D.

3.2. Static Compression Test Procedure

All tests were carried out in a 250 kN Instron testing machine under displacement control with the zero degree fibre direction parallel to the testing machine axis. The loading rate was approximately - 60 kN per minute. Due to the relatively short length available for gripping, the specimens were loaded into the test fixture shown in Fig. 2. The use of a core plate, brass shims and open-weave cloth-backed abrasive sheet (3M "Screenbak") enabled load transfer into the specimen by both end-loading and by shear along the faces of the specimen. The assembled test fixture was then gripped in the hydraulically operated grips of the testing machine. This technique proved to be satisfactory with all specimens failing near the centre of the gauge length without any evidence of buckling failures.

The specimens had a shorter gauge length than ideal and in view of the specimen thickness and loading arrangements it is likely that the near-surface plies of the coupon were more highly stressed than the central plies. However, no larger specimens could be obtained from the available material and since any stress gradient would be present in all of the specimens, the comparative testing approach used in this program was expected to be satisfactory.

3.3. Static Compression Test Results

3.3.1. Failure Strain

The static compression failure strain (Table 2a) for each coupon was calculated from a stress - strain calibration curve derived from earlier static tests on both 24 and 56-ply XAS-914C coupons.

Also shown in Table 2a are the sample means and standard deviations of static failure strain for each set of coupons.

3.3.2. Statistical Analysis

Shown in Table 2b are the results of a one-way analysis of variance of the coupon failure strains listed in Table 2a. The purpose of this analysis was to establish if there was any significant difference in compression strength between panels.

4. DISCUSSION OF TEST RESULTS

The test results for the impact-damaged fatigue test coupons (Table 1) show that for panel FG (which was not post-cured), there is a significant degradation (tested at a 95% confidence level) in fatigue performance. The mean test life for this panel is only 28.6 FLASTAFF blocks as against 80.5 and 91.3 FALSTAFF blocks for panels FH and FI.

In contrast, there is no significant difference in the static strength of material taken from these three panels, according to the results shown in Table 2. It appears from these results that complete curing of the epoxy matrix material can have a significant effect on the fatigue behaviour of impact-damaged CFC material, whilst having no significant effect on the static strength of undamaged material.

The additional polymer crosslinking completed by post curing must significantly enhance the ability of the matrix material to withstand fatigue damage development thereby delaying the onset of, or retarding, delamination growth.

The relationship between static strength and fatigue life is of considerable interest in the study of fatigue in composites; a number of strength-life ranking hypotheses suggest, for example, that the static strength ranking of a sample of testpieces matches (or may be related to) the ranking of those testpieces in fatigue life. It is important, however, to note that while the results presented here might appear to indicate little support for strength-life ranking, that conclusion must not be drawn - the appropriate comparison to evaluate strength-life ranking would involve testing damaged laminates under both static and fatigue conditions.

The results would seem to indicate that the mechanisms involved in (undamaged) static failure and (damaged) fatigue failure are different, at least in this particular material and layup. While further research is clearly required into the detailed mechanisms of damage growth and failure in composites, the results of this investigation are consistent with the view that impact damage growth involves a mechanism such as intra-ply cracking near the damage "tip", followed by linking of those intra-ply cracks by delaminations; in such a process, the mechanical (fracture) properties of the matrix material and/or matrix-fibre bonding would be expected to play a prominent role and hence state of cure should exert a significant influence. In the static case, however, failure is governed by the "weakest link" in a relatively large volume of material, and is more likely to be promoted by factors such as local fibre instability; failure should not therefore be greatly influenced by the relatively small changes in bulk matrix mechanical properties resulting from incomplete cure. At temperatures close to the glass transition temperature, and in particular in cases where the properties of the matrix are further degraded by the presence of moisture, a change to other failure modes is conceivable and it must be stressed that the present results are only applicable to room temperature loading conditions.

It is clear from this investigation that care may need to be exercised where compromise cure cycles may lead to incomplete matrix cure. Such incomplete curing can degrade laminate fatigue performance if the laminate is subjected to impact damage during service.

ACKNOWLEDGEMENT

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2. Royal Aeronautical Society Engineering Sciences Data Unit., Comparison of samples. Fatigue Sub Series Volume 2. Statistical methods Item No. 68016, London February 1968.

TABLE 1a

SPECIMEN FATIGUE LIVES - 30 mm DAMAGE IN FALSTAFF BLOCKS

SEQUENCE	PANEL FG		PANEL FH		PANEL FI	
Full FALSTAFF Sequence	FG2	7	FH7	67	FI6	34
	FG3	38	FH10	120	F18	77
	FG7	7.5	FH12	66	FI12	112
Truncated FALSTAFF Sequence	FG6	33.5	FH3	49	FI3	72
	FG8	42	FH8	56	FI7	123
	FG14	44	FH9	125	FI9	114
					FI14	107
Mean specimen life for each panel	28.6		80.5		91.3	
Standard deviation of panel test lives	17.0		33.2		31.8	

TABLE 1b

RESULTS OF TWO-WAY ANALYSIS OF VARIANCE

NULL HYPOTHESIS	CALCULATED VALUE OF F	LIMITING VALUE OF F (5% LEVEL)	CONCLUSION
No effect of sequence modification on fat. life	3.13	6.2	ACCEPT
No effect of panel-to-panel variation on fatigue life	10.61	4.8	REJECT

TABLE 2a

STATIC TEST COUPON COMPRESSION TEST FAILURE STRAINS

PANEL FG		PANEL FH		PANEL FI	
Coupon	Strain at failure (microstrain)	Coupon	Strain at failure (microstrain)	Coupon	Strain at failure (microstrain)
FG3A	-10 300	FH8B	-13 400	FI3A	-11 850
FG3B	11 950	FH9A	-11 300	FI3B	-10 700
FG3C	13 150	FH9B	-12 350	FI7A	-8 750
FG3D	11 100	FH12A	-12 500	FI7B	-12 550
FG7A	12 650	FH12B	-12 600	FI9	-11 400
FG7B	14 400	FH12C	11 000	FI11A	-12 000
FG7C	12 450	FH12D	-11 500	FI11B	-10 950
FG7D	11 700	FH14A	-13 750	FI14A	-12 500
FG8	12 350	FH14B	-12 800	FI14B	-11 550
FG14B	10 550				
Mean	12 060	Mean	-12 355	Mean	-11 361
Standard Deviation	1 233	Standard Deviation	935	Standard Deviation	1 1[illegible]2

TABLE 2b

RESULTS OF ONE WAY ANALYSIS OF VARIANCE

NULL HYPOTHESIS	CALCULATED VALUE OF F	LIMITING VALUE OF F (5% LEVEL)	CONCLUSION
No significant difference in static compression strength	2.59	4.3	ACCEPT

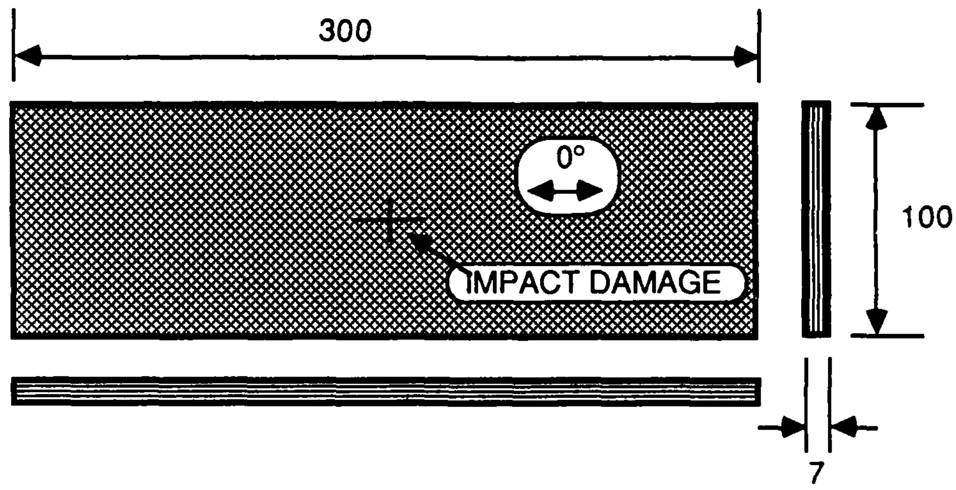

FIG 1a FATIGUE TEST COUPONS

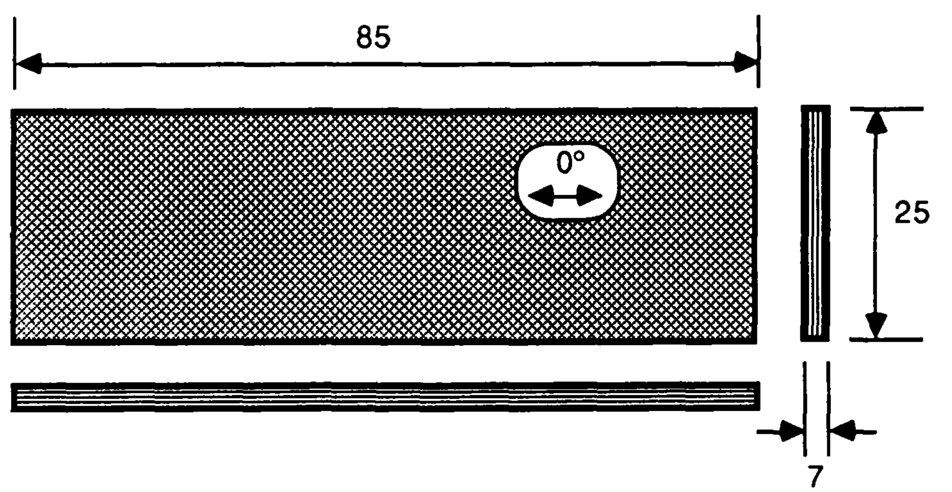

FIG 1b STATIC COMPRESSION TEST COUPONS

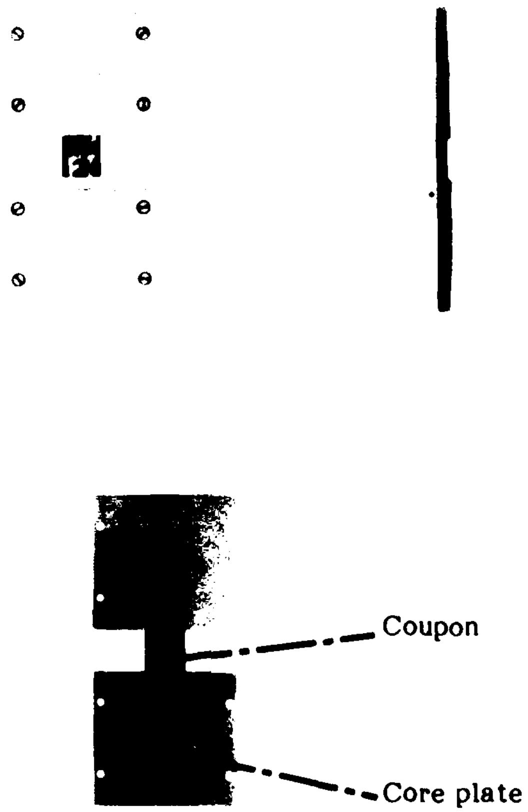

FIG 2 STATIC COMPRESSION TEST FIXTURES

APPENDIX I

(A) Cure Cycle For Ciba-Geigy XAS-914C Carbon Fibre Composite Preimpregnated Sheet.

(1) Vacuum at room temperature.

(2) Heat to 120^{o}C at 1 to 3^{o}C per minute.

(3) Hold at 120^{o}C for one hour.

(4) Pressurize autoclave to 700 kPa and vent vacuum.

(5) Raise temperature to 130^{o}C.

(6) Hold for 30 minutes at 130^{o}C.

(7) Raise temperature to 180^{o}C.

(8) Hold at 180^{o}C for one hour.

(9) Cool down to ambient or less than 60^{o}C with autoclave pressure held at 700 kPa before removal of laminate from autoclave.

(B) Post-cure for XAS-914C Laminate.

Post-cure in oven at 190^{o}C for four hours. Raise and lower temperature slowly.

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